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*Title:*

**OPERATIONAL AND REGULATORY  
PERFORMANCE OF WASTE CRATE ASSAY  
SYSTEMS AT RFETS**

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## **OPERATIONAL AND REGULATORY PERFORMANCE OF WASTE CRATE ASSAY SYSTEMS AT RFETS**

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### **ABSTRACT**

As Rocky Flats Environmental Technology Site (RFETS) approaches its closure target of 2006 emphasis for Non-Destructive Assay (NDA) has shifted from small waste package assay systems towards larger systems that are designed to accommodate Standard Waste Boxes (SWB) and larger Low Level Waste (LLW) containers. To this end, Kaiser Hill, with the support of BNFL Instruments, Inc. (BII) and Los Alamos National Laboratory (LANL), has recently deployed two new crate assay systems. These systems provide the capacity to meet the assay requirements associated with the Deactivation and Decommissioning (D&D) at RFETS.

The Super High Efficiency Neutron Coincidence Counting System (SuperHENC) was designed and fabricated as a collaborative effort between RFETS, LANL and BII. The purpose of this counter is to provide a WIPP certified assay capability for SWBs with a sensitivity that allows for TRU/LLW sorting. The SuperHENC has been in operation since early 2001.

The BII Multi-Purpose Crate Counter (MPCC) is based on the Imaging Passive Active Neutron (IPAN™) technology. This counter was designed to provide diverse capacity for WIPP certified assay of SWBs and to provide assay capability for larger LLW crates that are generated at RFETS. The MPCC has been in operation since early 2002.

In order to meet the requirement for measurement of the WIPP tracked radionuclides, both systems incorporate a BII Gamma Energy Analysis sub-system. The unique Energy Times Attenuation (ETA) method is used to provide isotopic mass fractions for diverse waste streams.

These systems were the first, and at this time the only, waste crate assay systems that have achieved WIPP certification. This represents a significant achievement given that the performance criteria applied to the measurements of large crates is identical to the criteria for 55-gallon (208 liter) drums. They are now both fully operational at RFETS and continue to successfully support the site closure mission.

## INTRODUCTION

As Rocky Flats Environmental Technology Site (RFETS) approaches its closure target of 2006 the requirements for Non-Destructive Assay (NDA) are changing. The emphasis has shifted from small waste package assay systems towards larger systems that are designed to accommodate Standard Waste Boxes (SWB) and larger Low Level Waste (LLW) containers. To this end, Kaiser Hill with the support of BNFL Instruments (BII) and Los Alamos National Laboratory (LANL) has recently deployed two new crate assay systems:

- The Super High Efficiency Neutron Coincidence Counting System (SuperHENC) a collaborative effort between RFETS, LANL and BII. This counter combines Neutron Coincidence Counting with Gamma Energy Analysis to assay SWBs with a sensitivity that allows for sorting at less than 100 nCi/g (3700 Bq/g). The system has been in operation since early 2001.
- The BII Multi-Purpose Crate Counter (MPCC) based on the Imaging Passive Active Neutron / Gamma Energy Analysis (IPAN™/GEA) technology. This crate counter provides diverse capacity assay of SWBs and larger LLW crates. The MPCC has operated since early 2002.

These assay systems provide the capacity to meet the assay requirements associated with the Deactivation and Decommissioning (D&D) at RFETS. Both assay systems have successfully completed certification for disposal to the Waste Isolation Pilot Plant (WIPP) including participation in the Performance Demonstration Program (PDP).

This paper summarizes the operational experience that has been gained during WIPP certification of these two assay systems and their continued operation in a production environment. The WIPP certification process was of particular interest not only to RFETS but also to the wider DOE weapons complex since these NDA systems were the first, and at this time the only, waste crate assay systems that have achieved WIPP certification.

## PERFORMANCE REQUIREMENTS

Both the MPCC and SuperHENC are required to identify and analyze the presence of plutonium, uranium, americium and other elements of interest in varying mass ratio concentrations in low, medium and high density waste matrices contained in various container sizes.

The systems must comply with WIPP and RFETS Nuclear Material Control and Accountability (MC&A) performance requirements for accuracy and precision (1,2). The same criteria as used for 55 gallon (208 liter) drum assays were selected as a conservative requirement due to the fact that no specific crate performance criteria have been promulgated to date.

### *WIPP Requirements*

For WIPP certification, the accuracy for the assay of a non-interfering matrix is not to exceed  $\pm 30\%$ . Precision (defined as %RSD) shall not exceed the values listed in Table I for the corresponding number of replicate measurements of a non-interfering matrix (1).

Table I. Upper Limits for %RSD vs. Number of Replicates

| No. Replicates | 3    | 4    | 5    | 6    | 7    | 8    | 9    | 10   | 11   | 12   | 13   | 14   | 15   |
|----------------|------|------|------|------|------|------|------|------|------|------|------|------|------|
| Max %RSD       | 6.60 | 10.0 | 12.3 | 14.0 | 15.2 | 16.2 | 17.1 | 17.7 | 18.3 | 18.8 | 19.3 | 19.7 | 20.0 |

In order to perform TRU/LLW sorting the systems must have a minimum detectable concentration (MDC) of less than 100 nCi/g (3700Bq/g).

### ***Nuclear Materials Control and Accountability (MC&A) Requirements***

The RFETS MC&A requirements for precision and accuracy (2) are summarized in Table II.

Table II. MC&A Quality Assurance Objectives for Radioassay

| Range of Pu and U (g) | Precision <sup>a</sup> (%RSD) | Accuracy <sup>b</sup> |
|-----------------------|-------------------------------|-----------------------|
| ≤1.0                  | 50                            | 50 to 150             |
| >1.0 to ≤10           | 25                            | 75 to 125             |
| >10.0                 | 10                            | 85 to 115             |

<sup>a</sup> Ratio of standard deviation in measured values of the known value, expressed as a percent

<sup>b</sup> Limits on the two-sided 95 percent confidence bound for the ratio of the mean of the measured values to the known value, expressed as a percent.

The accuracy requirement is particularly challenging for crate assay systems because allowance must be made for the measured precision i.e. the 95% confidence interval (derived from 2 tailed Student's T value) for the mean percent recovery (%R) must fall in the range given in Table II.

### ***Performance Demonstration Program (PDP) Requirements***

Both systems are required to participate in the PDP, a program used by Carlsbad Field Office (CBFO) as part of the assessment and approval of measurement facilities. PDP cycles comprise blind measurements of SWBs with surrogate matrices containing an unrevealed content of special nuclear material. The acceptance criteria in PDP measurements (3) are detailed in Table III. Note that the PDP pass criteria for SWB assays are identical to the 55 gallon (208 liter) drum criteria.

Table III. PDP Sample Activities and Associated Quality Assurance Objectives

| Range of waste activity in SWB (alpha-Curies) | Noninterfering matrix (%RSD) <sup>a</sup> | Interfering matrix (%RSD) <sup>a</sup> | Noninterfering matrix (%R) <sup>b</sup> | Interfering matrix (%R) <sup>b</sup> |
|---|---|--|---|--------------------------------------|
| >0 to 0.02                                    | 14  | 16                                     | 70 - 130                                | 40 - 160                             |
| >0.02 to 0.2                                  | 10.5                                      | 12                                     | 70 - 130                                | 40 - 160                             |
| >0.2 to 2.0                                   | 7   | 12                                     | 70 - 130                                | 40 - 160                             |
| >2.0  | 3.5                                       | 6                                      | 70 - 130                                | 40 - 160                             |

<sup>a</sup> Ratio of standard deviation in measured values of the known value, expressed as a percent

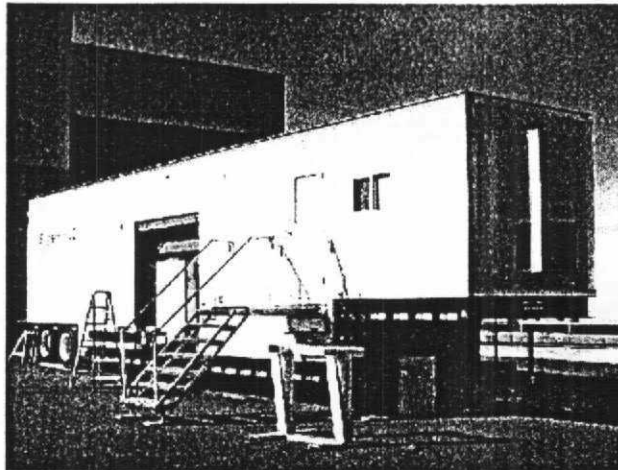
<sup>b</sup> Limits on the two-sided 95 percent confidence bound for the ratio of the mean of the measured values to the known value, expressed as a percent.

## **SuperHENC**

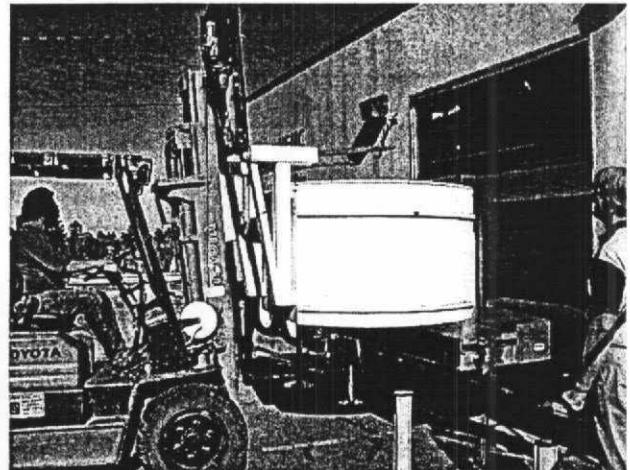
### ***System Description***

The SuperHENC is an entirely self-contained mobile system. It is mounted on a commercial 48 ft. (14 m) long "low boy" trailer that is 8 ft. 6 in. (2.6 m) wide and just under 13 ft. 6 in. (4.1m) high. The forward third of the trailer contains the control room. The middle third contains the SuperHENC itself. The aft section of the trailer has also been outfitted by BNFL Instruments Incorporated (BII) with a SuperHENC Gamma Energy Analysis System (SGEAS) for the

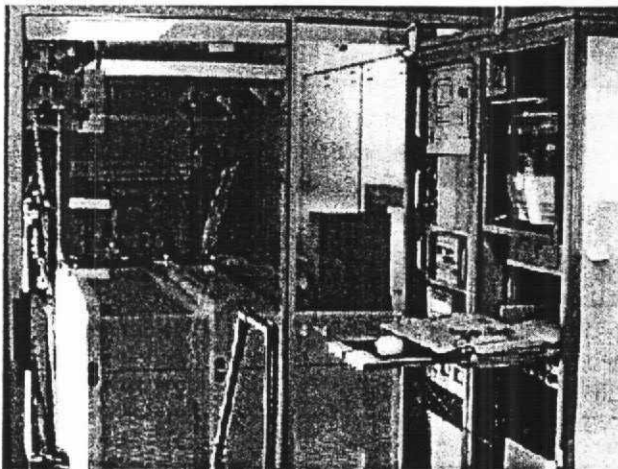
quantification of required radionuclides. The trailer is designed for rapid setup and leveling. Waste packages are loaded on to a fold down drawbridge with an integrated load cell and a motorized pallet to introduce the sample into the assay chamber. Figure 1 shows the SuperHENC trailer under operation.



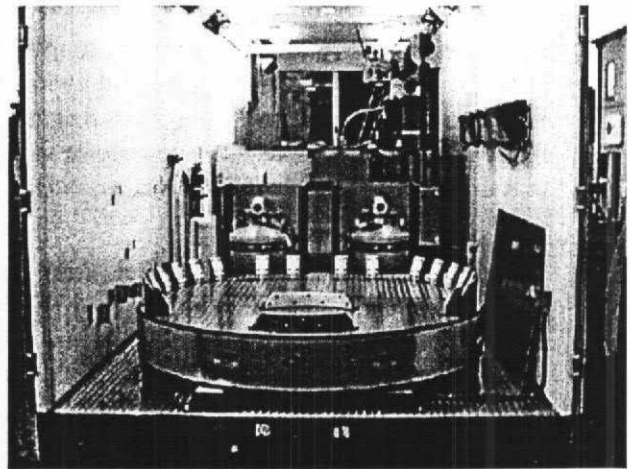
(a) System installed at RFETS site



(b) SWB loading into SuperHENC



(c) SuperHENC and Control Cabinet



(d) SGEAS gamma system

Fig. 1. Operation of the SuperHENC/ SGEAS trailer

The assay chamber is sized to accommodate at most an SWB with dimensions 65 in. (1.65 m) wide x 37 in. (0.94 m) high x 71 in. (1.80 m) long. It is designed to measure one SWB or one 55-gallon (208 liter) drum at a time. Figure 2 shows a diagram of the detector body with an SWB container inside the measurement cavity.

SuperHENC measures the  $^{240}\text{Pu}_{\text{eff}}$  content using passive neutron time-correlation counting and calculates the total plutonium content using Acceptable Knowledge (AK) for the plutonium isotopic mass fractions ( $^{238}\text{Pu}$ ,  $^{240}\text{Pu}$  and  $^{242}\text{Pu}$  are the only significant spontaneous fission source in the RFETS weapons grade Pu waste stream).

Top View of S-HENC

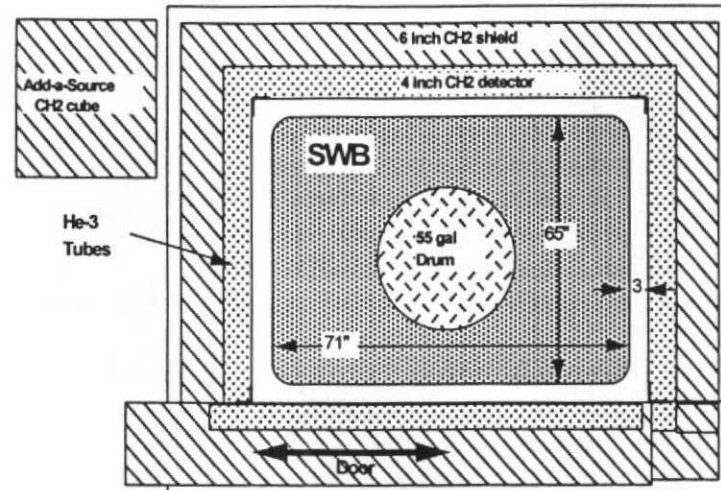


Fig. 2. Diagram of the SuperHENC detector body

The SuperHENC is operated by a custom tailored version of the LANL NCC software (4). The software controls container movement, data collection/analysis, and contains the calibration and error handling functions. After the passive neutron count, a small  $^{252}\text{Cf}$  add-a-source (AAS) (5) used for matrix correction is moved into the assay chamber automatically to pre-specified positions. After about a four-minute AAS count, the source is returned to its shielded storage area in the aft of the trailer. The chamber door opens, the package exits and is off loaded via fork truck. The entire assay system is constructed to minimize the presence of high atomic weight material; thereby minimizing the background from cosmic ray induced neutron cascades.

The package is then loaded on the SGEAS turntable for gamma measurement. The SGEAS software uses the BII Efficiency Times Attenuation technique, which is optimized for the sparse counting signal characteristic of large packages and dense matrices. The SGEAS software also automatically integrates the SuperHENC neutron data with the gamma data collected, and calculates the sample-specific MDC from the SuperHENC data. All WIPP reportable quantities such as TRU alpha activity concentration, decay heat and  $^{239}\text{Pu}$  fissile gram equivalent content are calculated by SGEAS. Automatic quality checks on the gamma result are also performed.

### Calibration

The SuperHENC measures the  $^{240}\text{Pu}_{\text{eff}}$  content using standard passive neutron doubles analysis. The calibration is based on an empty SWB doubles calibration curve with sample specific corrections applied for background and matrix effects.

The calibration was conducted in several steps. These steps included (i) constructing a Monte Carlo N-Particle (MCNP) model for the system, (ii) mapping chamber response with a neutron source, (iii) obtaining calibration measurements and establishing the coincidence calibration curve, (iv) establishing of the AAS correction factor calibration, (v) implementing background reduction techniques for samples with high backgrounds and low signals and (vi) validation of the calibration on different Pu standards at the final location (RFETS).

There is negligible multiplication over the plutonium mass range of interest and no neutron self-shielding (due to exclusive use of the passive mode), thus as expected, the calibration curve is simply a straight line through the origin.

Matrix specific corrections are handled in the count rate adjustment using the Add-A-Source (AAS) or the multiplicity matrix corrections. Complete equations for calculating final assay values may be found in the software documentation (4).

### ***Validation***

Validation measurements were collected on a non-interfering matrix (empty) standard for WIPP method performance demonstration, and on a variety of surrogate matrices that are representative of the waste stream at RFETS. Six replicates on three Pu loadings (1, 10, and 320 g) were collected. Sources were located in the approximate volume average position. This Pu range characterized the entire expected range, from near-MDC levels to the SWB criticality-loading limit. The matrix standards are described below, and a summary of the validation data presented.

The surrogate matrices comprise SWBs loaded with a modular matrix cube design representative of RFETS materials such as mixed metals, dry combustibles and plastics. Traditionally RFETS has segregated its waste streams into well defined "item description codes" (IDCs) such as mixed metals, dry combustibles and plastics. More recently, mixed matrix IDCs have been defined i.e.

- Inorganic matrices with less than 10% by weight of organics (IDC 3010)
- Inorganic matrices with greater than 10% by weight of organics (IDC 3011)

The benefit of looser segregation criteria is reduced human exposure, cost and difficulty and improved schedule. To meet MC&A qualification requirements for these new materials, mock up standards of IDC 3010 and 3011 were constructed by combining cubes of metal and plastics matrices to achieve 10% and 30% by weight organic content respectively.

Table IV gives a summary of the SuperHENC measurements taken at RFETS (6) on independent Pu standards to validate the calibration. Data was collected with the standard operating procedure and assay parameters used in routine operations. All of the measurements included in Table IV passed the applicable WIPP and MC&A data quality objectives for 55-gallon (208 liter) drums.

Table IV. Validation Measurement Summary for SWBs

| Matrix (IDC)                | 0.9g WG Pu |      | 9.0g WG Pu |      | 320g WG Pu       |                  |
|-----------------------------|------------|------|------------|------|------------------|------------------|
|                             | %R         | %RSD | %R         | %RSD | %R               | %RSD             |
| Metals (480)                | 140        | 6.4  | 109        | 4.1  | 102              | 0.9              |
| Mixed (3010)                | 110        | 3.5  | 94         | 1.0  | 99               | 0.8              |
| Mixed (3011)                | 92         | 4.0  | 95         | 0.8  | 102              | 1.1              |
| Plastics <sup>a</sup> (337) | N/A        | N/A  | 96         | 2.1  | 102              | 2.2              |
| Zero <sup>b</sup> (000)     | 126        | 13.4 | 106        | 1.5  | 103 <sup>b</sup> | 0.6 <sup>b</sup> |

<sup>a</sup> Plastics were not evaluated at the 0.9g level.

<sup>b</sup> The zero matrix box was tested at 160g.

### ***Lower Limit of Detection***

In accordance with the WIPP requirement, the SuperHENC's lower limit of detection (LLD) has been determined from background measurements at RFETS. Instruments performing TRU/LLW discrimination measurements must have an LLD or minimum detectable concentration (MDC) less than 100 nCi/g. MDC is defined as that radioactivity concentration which, if present, yields a measured value greater than the critical level with 95% probability, where the critical level is

defined as that value which measurements of the background will exceed with 5% probability. For the SuperHENC, MDC has been determined by statistical analysis of replicate assays of blank waste matrices (i.e containing no added activity).

The MDC must account for interferences from different matrix conditions and the on-site radiation background (dominated by the cosmic ray background). Additionally one must account for uncertainty that arises in quantifying the background component for an unknown sample using data taken with a surrogate matrix. It has been empirically determined (6) that a 15% uncertainty term sufficiently captures this uncertainty referred to as  $\sigma_F$ . This term is included in the MDC by adding it in quadrature to the standard deviation in the replicate assays.

Thus the critical level,  $L_C$ , is calculated as follows:

$$L_C = \bar{B} + \sqrt{(k \times \sigma_R)^2 + \sigma_F^2} \quad (\text{Eq. 1})$$

Detection limit,  $L_D$ , is therefore:

$$L_D = \bar{B} + 2 \times \sqrt{(k \times \sigma_R)^2 + \sigma_F^2} \quad (\text{Eq. 2})$$

and MDC is given by:

$$MDC = \frac{L_D \times Act_{spec}}{W_{net}} \quad (\text{Eq. 3})$$

where

- $\bar{B}$  is the average background corrected value from repeat assays of the blank,
- $k$  is a constant that relates to the one tailed normal distribution at 95% confidence,
- $\sigma_R$  is the standard deviation of the repeat assays of the blank matrix,
- $\sigma_F$  is the uncertainty due to matrix composition,
- $W_{net}$  is the net weight of the blank matrix,
- $Act_{spec}$  is the specific activity of the isotope or mixture of interest.

The MDCs are summarized in Table V for various surrogate matrices. These were calculated using RFETS weapons grade Pu isotopics (including approximately 30 year  $^{241}\text{Am}$  in-growth) and the net weight of the matrix. The minimum detectable activity (MDA) in terms of WGPU mass is also shown in Table V.

Table V. – Detection Limit Summary for the SuperHENC

| Matrix (IDC)           | Matrix weight (kg) | MDA (g WGPu) | MDC     |        |
|------------------------|--------------------|--------------|---------|--------|
|                        |                    |              | (nCi/g) | (Bq/g) |
| Empty (000)            | 0                  | 0.113        | N/A     | N/A    |
| Metals (480)           | 599                | 0.193        | 25.8    | 955    |
| Dry Combustibles (330) | 300                | 0.183        | 48.8    | 1806   |
| Mixed (3010)           | 535                | 0.219        | 29.2    | 1080   |
| Mixed (3011)           | 449                | 0.159        | 28.3    | 1047   |



### ***Performance Demonstration Plan (PDP) Results***

The first two box cycles of the SWB Performance Demonstration Testing was conducted in May 2001 and April 2002. A combustibles and a stainless steel matrix, were tested. Six replicates of each sample were taken using the SuperHENC/SGEAS system and integrated in accordance with the standard operating procedure. The final scoring results reported by the PDP (7,8) for these two cycles are presented in Table VI. All measurements passed the PDP SWB cycle acceptance criteria (3).

Table VI. SuperHENC PDP Cycles Summary for SWBs

| PDP<br>Box Cycle # | Measurement<br>Date | Matrix (IDC)       | Pu loading<br>(g WG Pu) | PDP Score |      |
|--------------------|---------------------|--------------------|-------------------------|-----------|------|
|                    |                     |                    |                         | %R        | %RSD |
| B1A                | May 2001            | Combustibles (330) | 7                       | 98        | 2.8  |
| B1A                | May 2001            | Metals (480)       | 10                      | 106       | 2.0  |
| B2A                | April 2002          | Combustibles (330) | 8                       | 91        | 5.2  |
| B2A                | April 2002          | Metals (480)       | 7                       | 100       | 2.7  |

### ***Operational Summary***

The SuperHENC/SGEAS has been successfully calibrated and validated at RFETS. All MC&A and WIPP criteria for assay of 55-gallon drums were met for the SWBs. Qualification of the new mixed-matrix waste streams crucial to the successful D&D of RFETS was demonstrated, achieving a new milestone in waste management at RFETS. PDP cycle data also met acceptance criteria. A detailed MDC assessment has been performed and demonstrates that the SuperHENC is capable of performing TRU/LLW sorting at the 100nCi/g level (3700 Bq/g). The combined SuperHENC/SGEAS system achieved WIPP certification and has been in full-time operation, measuring SWB waste at RFETS since early 2001.

## **MULTI-PURPOSE CRATE COUNTER**

### ***System Description***

The MPCC utilizes a polyethylene shielded assay cavity comprising three sections (See Figure 3). The front section houses the door assembly. The middle section houses the neutron detectors and neutron generator. The rear section houses the gamma detectors. The system accommodates crates up to 59 in. (1.50 m) wide x 59 in. (1.50 m) high x 91½ in. (2.32 m) long. A conveyor system runs the length of the cavity allowing a linear scan of a crate.

The four sides of the center section contain cadmium shielded <sup>3</sup>He detector packages that are sensitive to fast neutrons (see Figure 4). On one side, is the Zetatron neutron generator, housed within a moderator assembly (MA) constructed from lead, graphite and polyethylene.

The back section of the cavity is equipped with two liquid nitrogen cooled HPGe detectors one on each side of the cavity at different heights in order to accommodate the different sizes of crates (see Figure 4). A gamma filter array is provided for each detector, using various attenuating materials such as steel and copper to reduce gamma count rates for highly active crates.

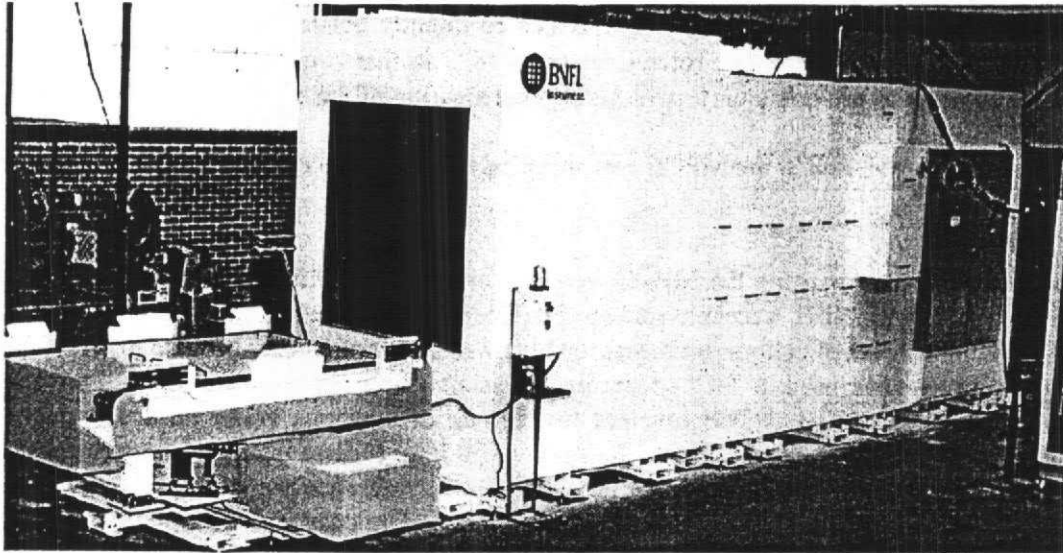


Figure 3. The MPCC at factory prior to installation in mobile enclosure.

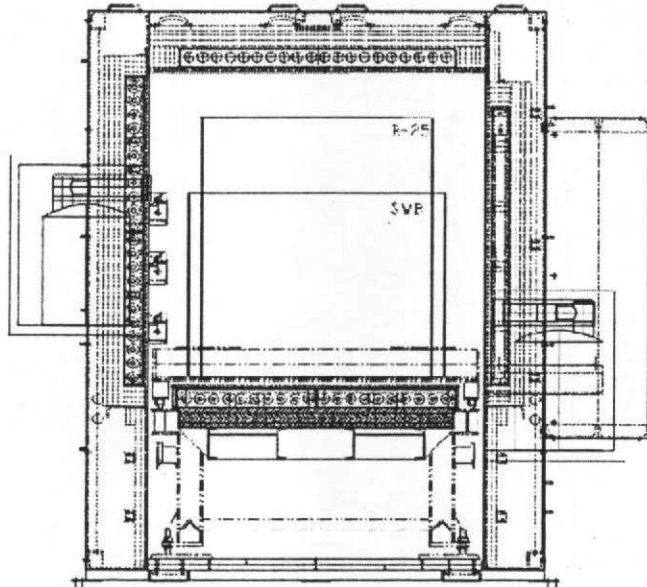


Figure 4. Cross sectional view of the MPCC

Two types of flux monitor detectors are installed in the MPCC: MA and Cavity flux monitors. The MA flux monitor comprises a bare  $^3\text{He}$  detector, located close to the neutron generator. This is used to monitor the magnitude of the interrogation flux produced by the neutron generator during the active measurement. Two types of cavity flux monitor detectors are installed inside the assay cavity on the side opposite the neutron generator. These detectors are used to quantify the absorption and moderation properties of the waste material in a crate. The two detector types are bare and shielded cavity flux monitors. A bare cavity flux monitor is a  $^3\text{He}$  detector that is installed within a cadmium collimator arrangement, which is collimated so that their field of view is limited to neutrons exiting from the waste crate. A shielded cavity flux monitor is a  $^3\text{He}$  detector that is installed within polyethylene that is completely shielded in cadmium. The two types of cavity flux monitors are configured in bare/shielded pairs at three heights on the side opposite the neutron generator making up three "dual" cavity flux monitors.

A turntable assembly that receives the crate is located directly outside the door in line with the cavity (see Figure 3). The turntable rotates the crate 180° so that two configurations of the crate are assayed in order to provide a uniform neutron interrogation of the crate.

For site installation at RFETS, the MPCC was installed into a mobile enclosure.

### ***Calibration***

Calibration measurements using the MPCC were performed in 1999 at the Arkansas factory and in 2002 at RFETS with NIST traceable sources (9). The calibrated Pu mass ranges from the MDC up to 320g WGPu. A set of calibration measurements was acquired using surrogate matrices. Two calibration sources were used: a  $^{252}\text{Cf}$  spontaneous fission source with an effective  $^{240}\text{Pu}$  mass of approximately 6.74g (at 3/15/02) equivalent to 114.78g of weapons grade Pu and 3 depleted uranium sources, each with an effective  $^{239}\text{Pu}$  mass of 500mg. Measurements were performed for various surrogate matrices with the source positioned at 36 reference points in each crate except for the SWB Empty where 12 reference points were used. The passive background rates in the Arkansas data were scaled to reflect the background coincidence rates at RFETS.

A technique for matrix selection in both active and passive mode making good use of the above calibration libraries has been developed for IPAN<sup>TM</sup> crate systems such as the MPCC. The flux monitor data from the active neutron interrogation is used to determine measured indices (known as ABSMOD and MOD). These are used to select the appropriate calibration matrix for each measurement. With this method, the calibration selection is predicated upon by the waste's neutronic properties. Thus the applicable calibration range is defined by the valid ranges in ABSMOD (for active mode) and MOD (for passive mode) and is not limited to the specific materials used in the calibration (e.g metals or plastics). Note that the MPCC software treats the B25 and SWBs as separate libraries so that a B25 may not be analysed as a SWB and vice-versa.

The threshold for active/passive neutron mode selection has been set at  $0.6\text{g }^{240}\text{Pu}_{\text{eff}}$  (i.e ~10g WG Pu). When the measured  $^{240}\text{Pu}_{\text{eff}}$  passive mass exceeds this level, the passive result is used to determine the total plutonium. Below this level the active mode is used.

### ***Validation***

Table VII provides a summary of the MPCC measurements taken at RFETS on independent Pu standards to validate the calibration. Data was collected with the standard operating procedure and assay parameters used in routine operations. All of the measurements included in Table VII passed the applicable WIPP and MC&A data quality objectives for 55-gallon (208 liter) drums.

Calculations using the MCNP code have been performed to determine the appropriate source correction terms to be applied to the lab mass due to artifacts of the test standards that are not normally encountered in real waste measurements (10). Accordingly, a correction term of 1.21 has been applied in order to yield an effective tag mass that accounts for absorption of interrogating thermal neutrons in the stainless steel encapsulation of the standard. No correction is required for passive mode measurements.

MPCC was tested against the RFETS MC&A validation requirements at the 0.9g, 9g and 320g Pu mass loadings for all of the required SWB calibration matrices (the empty SWB is validated in Section 5). The B25 matrices (LLW) were validated at the 0.9g loading. WIPP accuracy and precision requirements were demonstrated for the non-interfering (empty) calibration matrix.

Table VII. MPCC Validation Measurement Summary for SWBs

| Matrix (IDC)                            | 0.9g WG Pu |      | 9.0g WG Pu |      | 320g WG Pu       |                  |
|---|------------|------|------------|------|------------------|------------------|
|   | %R         | %RSD | %R         | %RSD | %R               | %RSD             |
| SWB Metals (480)                        | 103        | 2.9  | 92         | 0.4  | 99               | 3.8              |
| SWB Wet Combustibles (336)              | 109        | 0.9  | 83         | 0.7  | 105              | 3.1              |
| SWB Dry Combustibles (330)              | 121        | 2.2  | 108        | 0.3  | 106              | 4.0              |
| SWB Mixed <sup>a</sup> (3010)           | 99         | 5.1  | 92         | 0.3  | 87 <sup>a</sup>  | 4.4 <sup>a</sup> |
| SWB Mixed <sup>a</sup> (3011)           | 123        | 2.6  | 104        | 0.3  | 88 <sup>a</sup>  | 3.6 <sup>a</sup> |
| SWB Zero <sup>a</sup> (000)             | 102        | 1.4  | 96         | 0.8  | 103 <sup>a</sup> | 3.3 <sup>a</sup> |
| B25 Wet Combustibles <sup>b</sup> (336) | 102        | 0.9  | N/A        | N/A  | N/A              | N/A              |
| B25 Dry Combustibles <sup>b</sup> (330) | 109        | 1.2  | N/A        | N/A  | N/A              | N/A              |
| B25 Metals <sup>b</sup> (480)           | 108        | 1.2  | N/A        | N/A  | N/A              | N/A              |

<sup>a</sup> The zero matrix box and mixed matrices were tested at 160g.

<sup>b</sup> B25 boxes are a LLW stream and therefore only evaluated at the 0.9g level

### Lower Limit of Detection

In an IPAN<sup>TM</sup> system, the active mode detection limit is several orders of magnitude more sensitive than the passive mode detection limit. <sup>239</sup>Pu<sub>eff</sub> mass is derived from the fissile signal. Using the same methodology for MDC determination as described above for SuperHENC, a set of replicate blank measurements were made for each SWB calibration matrix. The baseline fissile signal detection limit,  $FS_{LD,0}$  is given by

$$FS_{LD,0} = \frac{j \sqrt{\left(1 + \frac{1}{n}\right) \left(S_{EG,0}^2 + a^2 S_{LG,0}^2\right)}}{\overline{t'_0}} \quad (\text{Eq. 4})$$

where

- $\overline{t'_0}$  is the average original neutron generator flux for the blank measurements,
- $j$  is a constant that relates to the one tailed normal distribution at 95% confidence,
- $n$  is the number of blank measurements,
- $S_{EG,0}$  and  $S_{LG,0}$  are the standard deviations in specific gated counts for the blanks,
- $a$  is a ratio of gate lengths in active mode acquisition.

For each individual waste measurement, the neutron generator flux will differ from the baseline flux. To first order approximation, the fissile signal detection limit is:

$$FS_{LD} = FS_{LD,0} \sqrt{\left(f + \frac{1}{n}\right) \left(\frac{n}{n+1}\right)} \quad (\text{Eq. 5})$$

where  $f$  is the ratio of the original neutron generator flux to the new flux.

The baseline MDC is determined analogous to the manner described for SuperHENC (Eq.3):

$$MDC_0 = \frac{K_1 FS_{LD} Act_{spec}}{W_{net}} \quad (\text{Eq. 6})$$

where  $K_1$  is a matrix dependent factor derived from the calibration data. This is the ratio of net fissile signal to Pu mass based on a volume-weighted average over all calibration positions. The baseline MDA (g WG Pu) and MDC are summarized in Table VIII for various surrogate SWBs.

Table VIII. Detection Limit Summary for the MPCC IPAN™/GEA.

| Matrix (IDC)                 | Matrix weight (kg) | Baseline MDA (g WGPu) | Baseline MDC |        |
|------------------------------|--------------------|-----------------------|--------------|--------|
|                              |                    |                       | (nCi/g)      | (Bq/g) |
| Empty SWB <sup>a</sup> (000) | 136                | 0.018                 | 10.0         | 370    |
| Metals SWB (480)             | 575                | 0.029                 | 3.8          | 141    |
| Dry Combustibles SWB (330)   | 305                | 0.048                 | 11.8         | 437    |
| Mixed SWB (3010)             | 531                | 0.108                 | 15.4         | 569    |
| Mixed SWB (3011)             | 436                | 0.083                 | 14.5         | 536    |

<sup>a</sup> A nominal matrix mass of 136 kg was used to defined the empty SWB MDC

Under routine operations, sufficient neutron generator output flux must be maintained to ensure that the detection limit is below 100 nCi/g (3700 Bq/g). To achieve this the generator output as measured by the MA flux monitor (MAFM) must meet the following conditions.

$$MAFM > \frac{\overline{MAFM}_0}{\frac{(n+1)}{n} \left( \frac{100}{MDC_0} \right)^2 - \frac{1}{n}} \quad (\text{Eq. 7})$$

where  $\overline{MAFM}_0$  is the baseline MAFM counts

For all matrices, the resulting minimum flux is at least an order of magnitude less than the baseline flux, thus this condition can easily be achieved under normal operating conditions.

### **Performance Demonstration Plan (PDP) Results**

The first two box cycles of the SWB Performance Demonstration Testing was conducted in April 2002 (8). A combustibles and a stainless steel matrix, were tested. Six replicates of each sample were taken using the MPCC system and integrated in accordance with the standard operating procedure. Final results are presented in Table IX. All measurements passed the PDP SWB cycle acceptance criteria (3).

Table IX. MPCC PDP Cycle Summary for SWBs

| PDP<br>Box Cycle # | Measurement<br>Date | Matrix (IDC)       | Pu loading<br>(g WG Pu) | PDP Score |      |
|--------------------|---------------------|--------------------|-------------------------|-----------|------|
|                    |                     |                    |                         | %R        | %RSD |
| B2A                | April 2002          | Combustibles (330) | 8                       | 86        | 3.7  |
| B2A                | April 2002          | Metals (480)       | 7                       | 80        | 1.8  |

### ***Operational Summary***

The MPCC IPAN™/GEA assay systems is now fully qualified for WIPP measurements, and has demonstrated TRU/LLW sorting capability at the 100nCi/g (3700 Bq/g) level. The system achieved the PDP pass criteria in the 2002 cycle. With the exception of IDC 3011 at 320g, the MC&A criteria for new mixed-matrix waste streams were demonstrated. The system has been in operation since early 2002.

### **SUMMARY**

The SuperHENC/SGEAS and the MPCC IPAN™/GEA assay systems are now qualified for MC&A measurements, certified for WIPP measurements, and are performing waste box assays with a demonstrated TRU/LLW sorting capability at the 100nCi/g (3700 Bq/g) level. Qualification of the new mixed-matrix waste streams crucial to the successful D&D of RFETS was demonstrated.

For the first time, PDP cycles for box assay have been performed, and both systems have met all of the required criteria.

The SuperHENC developed by LANL, BII and RFETS, has demonstrated excellent performance results for SWBs over a diverse calibration range.

The MPCC supplied by BII, vastly expands the site crate capability as it can accommodate waste boxes up to 11,000 pounds and can measure both SWBs and B25s within the calibrated range.

Both box assay systems have established significant milestones in achieving WIPP and MC&A certification, especially given that they met the data quality objectives that were originally developed for 55 gallon (208 liter) drums.

### **REFERENCES**

1. Contact Handled Transuranic Waste Acceptance Criteria, DOE/WIPP-02-3122, rev 0.1, July 25 2002, U.S. DoE Carlsbad Field Office
2. MAN-010-MCA Nuclear Materials and Accountability Manual, RFETS
3. Performance Demonstration Program Plan for Non-Destructive Assay of Boxed Wastes for the TRU Waste Characterization Program, DOE/CBFO-01-1006 Rev. 0, January 31 2001, U.S. DoE Carlsbad Field Office
4. W. Harker and M. Krick, "Mobile, High Efficiency Neutron Counter Software Design Document," Los Alamos National Laboratory document LA-CP-00-446.

WM'03 Conference, February 23-27, 2003, Tucson, AZ

5. H. O. Menlove, "Passive Neutron Waste Drum Assay with Improved Accuracy and Sensitivity for Plutonium Using the Add-a-Source Method," *JNMM* 17, pp. 17-26 (July 1992).
6. H.O Menlove, et al, SuperHENC: Final Performance And Certification Summary, LA-UR-01-6459, Environmental Management Nondestructive Assay Characterization Conference, Denver, Colorado, December 11-13, 2001
7. Performance Demonstration Program Plan for Non-Destructive Boxed Assay, Scoring Report – Cycle B1A, May 2001 Distribution, Carlsbad Field Office Technical Assistance Contractor
8. Performance Demonstration Program Plan for Non-Destructive Boxed Assay, Scoring Report – Cycle B2A, April 2002 Distribution, Carlsbad Field Office Technical Assistance Contractor
9. Simpson A.P., RFETS MPCC Imaging Passive Active Neutron and Gamma Energy Analysis System Calibration Test Report, BNFL Instruments Report, Santa Fe, NM, BII-8197-CTR-001, Rev 1, April 2002
10. G Auchampaugh, "Monte Carlo Assessment of Correction Factors in Pu Verification Standards for RFETS 569 Verification", BNFL Instruments Report, Los Alamos, NM, March 2000, BII-8215-TN99-003, rev 1